Effects of annealing cooling rates on mechanical properties, microstructure and texture in continuous annealed IF steel

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Effects of annealing cooling rates on mechanical properties, microstructure and texture in continuous annealed IF steel

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Abstract

The mechanical properties, microstructure and texture of interstitial-free (IF) steel processed through continuous annealing routes under five cooling rates (5 °C/s, 50 °C/s, 200 °C/s, 500 °C/s and 1000 °C/s) have been studied. The cooling rates of 200 °C/s, 500 °C/s and 1000 °C/s are defined as ultra-fast cooling rates. This paper focuses on the differences of mechanical properties, microstructure and texture evolution between ultra-fast and common cooling rates (5 °C/s, 50 °C/s) for the first time. The overall structural features of specimens were observed by optical microscopy and nano-sized precipitates were observed by transmission electron microscope (TEM) on carbon extraction replicas. Texture measurement was carried out using D8 advance X-Ray Diffraction (XRD) and the (110), (200) and (112) pole figures were determined. The yield strength increases with increasing cooling rates, from 96 MPa under the cooling rate of 5 °C/s to 136 MPa under the cooling rate of 1000 °C/s, while the ultimate tensile strength changes very little (around 280 MPa). The samples annealed under the cooling rates of 5 °C/s and 500 °C/s have low r_{ave} values (2.13 and 2.21 respectively). By contrast, the samples in cases of 50 °C/s, 200 °C/s and 1000

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C/s possess higher $r_{ave}$ values (2.32, 2.40 and 2.51 respectively). Three morphologies of TiN particles can be observed in all samples. TiN, with CaO and SiO$_2$ acting as heterogeneous nuclei, is prone to have big size. The size of TiN precipitates decreases as increasing of cooling rates. FeTiP is found near TiC precipitates under the cooling rate of 5 °C/s. The sample annealed under the cooling rate of 5 °C/s has the weakest $\{111\} <112>$ and $\{111\} <110>$ orientations. The intensity of $\{111\} <110>$ component increases as the cooling rate increases from common cooling rates of 5 °C/s and 50 °C/s to ultra-fast cooling rates of 200 °C/s, 500 °C/s and 1000 °C/s. The intensity of $\{011\} <100>$ orientation decreases as the cooling rate increases from 5 °C/s to 500 °C/s. The relationship between $r$ value, microstructure and texture components has been studied in this paper.

**Key Words:** Interstitial-free steel; Precipitation; Texture; Cooling rates; Continuous annealing

1. Introduction

Application of IF steels in the automotive industry has seen a significant increase over the past couple of decades due to their excellent deep-drawing property[1]. Titanium (Ti) and niobium (Nb) are added to these steels, singly or in combination, to scavenge carbon and nitrogen by the formation of carbides and nitrides and thus remove interstices from the matrix[2-4]. This clear matrix is essential for increased formability and is suitable for galvanneal coating, which is required for automotive body and home appliances[5]. The main trends in the development of IF steels are
towards high-strength and high-formability varieties. \textcolor{green}{P and Mn have been added in IF steels as solution strengthening elements to improve strength performance.}

Precipitation behavior in IF steels is quite complex and TiN, TiS, TiC and NbC precipitates are quite common in Ti-IF steels[6-8]. Characterization of TiN, TiC and Ti (C, N) in titanium-alloyed ferritic chromium steels focusing on different particle morphologies has been studied by Michelic[9]. A detailed analysis of TiN morphology and composition shows that there are four subcategories, pure TiN, TiN with an oxide nuclei, TiN with a carbide or carbonitride layer and TiN clusters (agglomerated single TiN), which differ significantly in size and morphology in ferrite stainless steels[9]. Ghosh and his group[10] have studied the thermodynamics of precipitation and textual development in IF steels. Above 900 °C, the TiCN phase mainly contains N apart from Ti, which is simply TiN. However, at around 900 °C, independent TiC formation is likely to take place and the amount of carbon is considerably increased in this phase below 900 °C[10]. The precipitation behavior of FeTiP-type precipitates in IF steels has been studied[2-4, 11]. P has been added to normal IF steels as solid solution strengthening elements and this leads to the formation of FeTiP precipitates in annealing process. \textcolor{green}{FeTiP prevents the growth of \{111\} recrystallization texture.}

FeTiP usually precipitates at the grain boundary and the segregation of phosphorus atoms at grain boundaries has been studied before[12]. During the annealing process, non-equilibrium segregation of phosphorus atoms at the grain boundaries is related to the moving dislocation lines[12-14].
The strong \{111\} recrystallization textures are beneficial for deep-drawing property and their formation mechanism and texture evolution have been studied in several literatures\cite{15-20}. Path of crystal rotation during rolling and the impacts of alloying elements and second phase particles on texture formation in low carbon steels have been studied\cite{21}. It is reported that the precipitates increase the recrystallization temperature and hinder the recrystallization texture\cite{22}. In addition, different texture components have different impacts on $r$ and $\Delta r$ values. The \{011\} <001> component contributes to the increase of $r$ value greatly in BCC materials, but it also increases $\Delta r$ value. The \{111\} <110> and \{111\} <112> components can increase $r$ value without increasing $\Delta r$ value\cite{23, 24}.

Previous studies focused on mechanical properties and microstructure under different continuous annealing temperature and holding time. As for cooling rates, few studies have been made, especially under ultra-fast cooling rates. The present industrial production continuous annealing cooling rate is lower than 50 °C/s and precipitation behavior and texture evolution under ultra-fast cooling rates have not been studied. While apart from common cooling rates (5 °C/s and 50 °C/s), ultra-fast cooling rates (200 °C/s, 500 °C/s and 1000 °C/s) were also carried out in this experiment. This paper makes comparison studies between effects of common cooling rates (5 °C/s and 50 °C/s) and ultra-fast cooling rates (200 °C/s, 500 °C/s and 1000 °C/s) on mechanical properties (focusing on $r$ values), microstructure and texture components. Considering the low $r_{ave}$ values in cases of 5 C/s and 1000 C/s and their different major texture components, a relationship between the $r$ value, $\Delta r$ value and
texture components in IF steel has been established. By controlling annealing process, the preferred texture components, which increase $r$ value and decrease $\Delta r$ value, can be obtained. Thus the formability of the material can be improved. Studying different continuous annealing cooling rates also supplies the IF continuous annealing studies and improves continuous annealing efficiency.

2. Experimental and analytical methods

The starting material was a Ti-IF steel of 1 mm in thickness and its chemical compositions are listed in Tab.1. P has been added as solid solution strengthening elements. The steel was prepared by hot rolling and cold rolling and the processing parameters are given in Tab.2.

<table>
<thead>
<tr>
<th>Tab. 1 Chemical composition of the IF steel (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
</tr>
<tr>
<td>0.0015</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Tab. 2 Processing parameters of the IF steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot finish rolling temperature ($^\circ$C)</td>
</tr>
<tr>
<td>930</td>
</tr>
</tbody>
</table>

There were five stages in the annealing cycles: (1) HS (heating section): heating from room temperature to soaking temperature ($810 ^\circ$C); (2) SS (soaking section): holding at soaking temperature for 180 s; (3) CS (cooling section): cooling to ambient temperature (cooling rates range from 5 $^\circ$C/s to 1000 $^\circ$C/s); (4) OA (over aging section): holding at overaging temperature ($240 ^\circ$C) for 120 s; (5) AC (air cooling): air cooling to room temperature. The continuous annealing process was simulated in the CCT-AY heat treatment system under the cooling rates of 5 $^\circ$C/s and 50 $^\circ$C/s. As for
the cooling rates of 200 °C/s, 500 °C/s and 1000 °C/s, the steels were cooled in different mediums. The different annealing tests were performed on laboratory scale. Ultra-fast cooling rates could be applied on industrial scale and the Kobe Steel Group Japan has already applied ultra-fast cooling rates on steel production. The continuous annealing curve is shown in Fig.1.

![Continuous annealing curve of the IF steel](image)

Fig.1 Continuous annealing curve of the IF steel

The mechanical properties of the samples annealed under different cooling rates, including yield strength (YS), ultimate tensile strength (UTS), elongation, n and r values were investigated at room temperature in three directions of 0°, 45° and 90° in the tension test. Further TEM equipped with an energy dispersive spectrometer (EDS) was used to study the precipitation behavior. Specimens for TEM were prepared by carbon extraction replicas. Macroscopic textures were measured on an X-ray diffractometer and three pole figures {110}, {200}, and {112} were obtained. Orientation distribution functions (ODFs) were calculated from these pole figures using the Bunge method[25]. The α, γ, η, ζ, 0, and ε-fiber intensities were also plotted in this study.

3. Results
3.1 Mechanical properties under different cooling rates
It can be clearly seen that their mechanical properties (shown in Tab.3) are different under different cooling rates. The yield strength turns out gradually upward trend with the increase of cooling rates, while the ultimate tensile strength changes little (around 280 MPa). The yield ratio increases with the increase of cooling rates. The yield ratio is the ability to resist deformation from yielding to plastic instability and the lack of adequate strain hardening results in high yield ratio\cite{26, 27}. The mechanical results also reveal that the cooling rates have little effect on elongation and $n$ values.

The $r$ value is defined as true plastic strain ratio of width to thickness under uniaxial tension and low $r$ value indicates poor deep-drawing property \cite{28}.

\[ r_{ave} = \frac{(r_0 + 2r_{45} + r_{90})}{4} \]  
\[ \Delta r = \frac{(r_0 - 2r_{45} + r_{90})}{2} \]

The $r_{ave}$ values of specimens annealed under the cooling rates of 5 °C/s and 500 °C/s are significantly lower than others. The $r_{ave}$ value is 2.40 when the cooling rate is 200 °C/s and it decreases to 2.21 under the cooling rate of 500 °C/s.

In addition, LDR (limiting drawing ratio) value is usually used to evaluate deep drawability and theoretically can be expressed as follows:

\[ LDR = \sqrt{\exp \left[ 2f \exp(-n) \right] \frac{(1+r)}{2} + \exp \left[ 2n \sqrt{\frac{(1+r)}{2}} \right] - 1} \]

Where $f$ is the factor of drawing efficiency, and when $f$ equals to 0.9, the calculated results are in good agreement with the experimental results\cite{26}. Based on the equation, the LDR values of samples annealed under different cooling rates (form 5 °C/s to 1000 °C/s) are 2.53, 2.59, 2.61, 2.18 and 2.22 respectively. This result reveals
that the deep property of sample annealed under the cooling rate of 200 °C/s is higher than others.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Cooling rate (°C/s)</th>
<th>Direction</th>
<th>$r_{\text{ave}}$</th>
<th>$\Delta r$</th>
<th>$n_{\text{ave}}$</th>
<th>Elongation (%)</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
</tr>
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<tr>
<td>1#</td>
<td>5</td>
<td>45</td>
<td>1.72</td>
<td>0.81</td>
<td>0.31</td>
<td>0.30</td>
<td>52.4</td>
<td>96</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>2.66</td>
<td>0.31</td>
<td>0.30</td>
<td>54</td>
<td>100</td>
<td>271</td>
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<tr>
<td></td>
<td>0</td>
<td>2.83</td>
<td>0.30</td>
<td>0.30</td>
<td>53.5</td>
<td>103</td>
<td>280</td>
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</tr>
<tr>
<td>2#</td>
<td>50</td>
<td>45</td>
<td>1.82</td>
<td>2.32</td>
<td>1</td>
<td>0.30</td>
<td>50</td>
<td>105</td>
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<tr>
<td></td>
<td>90</td>
<td>2.81</td>
<td>0.31</td>
<td>0.30</td>
<td>48</td>
<td>102</td>
<td>276</td>
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<tr>
<td></td>
<td>0</td>
<td>2.93</td>
<td>0.30</td>
<td>0.30</td>
<td>50.2</td>
<td>119</td>
<td>277</td>
<td></td>
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<tr>
<td>3#</td>
<td>200</td>
<td>45</td>
<td>1.94</td>
<td>2.40</td>
<td>0.91</td>
<td>0.29</td>
<td>0.30</td>
<td>49.3</td>
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<td>2.77</td>
<td>0.30</td>
<td>0.30</td>
<td>51.2</td>
<td>118</td>
<td>277</td>
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<tr>
<td></td>
<td>0</td>
<td>2.53</td>
<td>0.30</td>
<td>0.30</td>
<td>50.3</td>
<td>123</td>
<td>284</td>
<td></td>
</tr>
<tr>
<td>4#</td>
<td>500</td>
<td>45</td>
<td>1.84</td>
<td>2.21</td>
<td>0.75</td>
<td>0.29</td>
<td>0.29</td>
<td>47.6</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>2.64</td>
<td>0.29</td>
<td></td>
<td>48</td>
<td>127</td>
<td>280</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0</td>
<td>3.03</td>
<td>0.30</td>
<td>0.30</td>
<td>51.6</td>
<td>126</td>
<td>275</td>
<td></td>
</tr>
<tr>
<td>5#</td>
<td>1000</td>
<td>45</td>
<td>2.02</td>
<td>2.51</td>
<td>0.97</td>
<td>0.29</td>
<td>0.29</td>
<td>48.6</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>2.96</td>
<td>0.28</td>
<td></td>
<td>48</td>
<td>132</td>
<td>277</td>
<td></td>
</tr>
</tbody>
</table>

3.2 Microstructure under different cooling rates

3.2.1 Grain sizes

The optical micrographs of IF steels annealed under different cooling rates are presented in Fig.2 and the effect of cooling rates on the ferrite grain sizes is shown in Fig.3. All samples are made up of equiaxed ferrite grains. The results show that grain sizes decrease with increasing cooling rates. The mean grain size reaches its maximum (15.13 µm) under the cooling rate of 5 °C/s and decreases to the minimum value of 11.75 µm under the cooling rate of 1000 °C/s. According to statistics calculation, the average grain aspect ratio (17:10) in steels under the cooling rates of 5 °C/s and 50 °C/s is a little higher than that (about 3:2) in steels annealed under the ultra-fast cooling rates of 200 °C/s, 500 °C/s and 1000 °C/s.
Fig. 2 Optical micrographs of IF steels under different cooling rates (a) 5 °C/s; (b) 50 °C/s; (c) 200 °C/s; (d) 500 °C/s; (e) 1000 °C/s
3.2.2 Precipitation behavior

Fig. 4 shows the presence of precipitates in samples annealed under different cooling rates. A general observation made from the TEM micrographs is that cuboid TiN is the most common precipitates under all different cooling rates and TiC precipitates are in elliptical shape. Three morphologies of TiN precipitates: (1) pure TiN; (2) TiN clusters (Fig. 4d); (3) TiN with an oxide nuclei (Fig. 5); can be observed. TiN particles (with oxide nuclei), which grew under the cooling rates of 5 °C/s and 50 °C/s, are greatly larger than those grown under ultra-fast cooling rates. The sizes of pure TiN observed under different cooling rates are shown in Tab. 4. and they decrease with increasing cooling rates. Fig. 6 reveals elliptical TiC precipitates and the presence of FeTiP which formed on the pre-existing TiC particle under the cooling rate of 5 °C/s only. The schematic development (including morphologies and forming stage) of different precipitates is shown in Fig. 7.
Fig. 4 TEM extraction replica micrographs showing presences of different precipitates under various cooling rates
(a) 5 °C/s; (b) 50 °C/s; (c) 200 °C/s; (d) 500 °C/s; (e) 1000 °C/s; (f) non-annealed sample
Fig. 5 TEM micrographs showing the presence of large size TiN precipitates formed under the cooling rates of 5 °C/s (a) and 50 °C/s (b) and corresponding EDS spectra (c) and (d).

| Tab. 4 Different sizes of pure TiN precipitates under different cooling rates |
|-------------------------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Cooling rates/ °C·s⁻¹ | Common cooling rates | Ultra-fast cooling rates | Non-annealed sample |
|----------------------|------------------|------------------|------------------|------------------|------------------|
| TiN sizes/nm         | 25~25            | 20               | 10~15            | 10~15            | 10~12            | 10               |
Fig. 6 TEM extraction replica micrographs showing presents FeTiP and TiC precipitates formed under the cooling rate of 5 °C/s (a) and corresponding EDS spectra FeTiP (b) and TiC (c).

Fig. 7 Schematic development of various morphologies of different precipitates over different process.

3.3 Texture evolution under different cooling rates

The ideal orientations of texture components in BCC materials are shown schematically for $\varphi_2=0^\circ$ and $45^\circ$ ODF sections in Fig.8 and Tab.5. For IF steel, the vital fibers are the $\alpha$-fiber (fiber axis $<110>$ parallel to the rolling direction including components of $\{001\}$ $<110>$, $\{112\}$ $<110>$ and $\{111\}$ $<110>$), $\gamma$-fiber (fiber axis $<111>$...
parallel to the normal direction including components of \{111\} \langle110\> and \{111\} \langle112\>, \(\varepsilon\)-fiber (fiber axis \langle011\> parallel to the transverse direction including components of \{001\} \langle110\>, \{112\} \langle111\>, \{111\} \langle112\> and \{011\} \langle100\>), \(\eta\)-fiber (fiber axis \langle100\> parallel to the rolling direction including components of \{001\} \langle100\> and \{011\} \langle100\>), \(\theta\)-fiber (fiber axis \langle001\> parallel to the normal direction including components of \{001\} \langle100\> and \{001\} \langle110\> ) and \(\xi\)-fiber (fiber axis \langle011\> parallel to the normal direction including components of \{011\} \langle100\>, \{011\} \langle211\>, \{011\} \langle111\> and \{011\} \langle011\>[18].

![Fig. 8 Schematic illustration of the important texture components in BCC materials](image_url)

(a) \(\phi_2=0^\circ\) (b) \(\phi_2=45^\circ\)
Tab. 5 The important fibers and orientations for BCC materials

<table>
<thead>
<tr>
<th>Fiber name</th>
<th>Fiber axis</th>
<th>Important components</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\alpha)</td>
<td>(&lt;110&gt;/&gt;RD)</td>
<td>(&lt;001&gt;&lt;110&gt;, [112]&lt;110&gt;, [111]&lt;110&gt;)</td>
</tr>
<tr>
<td>(\gamma)</td>
<td>(&lt;111&gt;/&gt;ND)</td>
<td>(&lt;111&gt;&lt;110&gt;, [111]&lt;112&gt;)</td>
</tr>
<tr>
<td>(\epsilon)</td>
<td>(&lt;011&gt;/&gt;TD)</td>
<td>(&lt;001&gt;&lt;110&gt;, [112]&lt;111&gt;, [111]&lt;112&gt;, [011]&lt;100&gt;)</td>
</tr>
<tr>
<td>(\eta)</td>
<td>(&lt;100&gt;/&gt;RD)</td>
<td>(&lt;001&gt;&lt;100&gt;, [011]&lt;100&gt;)</td>
</tr>
<tr>
<td>(\theta)</td>
<td>(&lt;001&gt;/&gt;ND)</td>
<td>(&lt;001&gt;&lt;100&gt;, [001]&lt;110&gt;)</td>
</tr>
<tr>
<td>(\xi)</td>
<td>(&lt;011&gt;/&gt;ND)</td>
<td>(&lt;011&gt;&lt;100&gt;, [011]&lt;211&gt;, [011]&lt;111&gt;, [011]&lt;011&gt;)</td>
</tr>
</tbody>
</table>

Fig. 9 illustrates the ODFs of samples annealed under different cooling rates. Changes of maximum intensity versus the annealing cooling rates for major texture components are presented in Fig. 10. It can be seen that the main texture orientations were \{111\} \(<112>, [111]<110>\> and \{011\} \(<100>\> with the maximum intensity of 4.09×R, 4.15×R and 2.59×R when the cooling rate was 5 °C/s. Fig. 9(b) indicates that the intensity of \{111\} \(<112>\> orientation increased by 25%, from 4.09×R to 5.06×R, under the cooling rate of 50 °C/s. There was also an increase in the intensity of \{001\} \(<110>\> orientation from 0.453×R to 1.54×R. It should be noted that the intensity of \{111\} \(<110>\> increased by 21%, from 4.49×R to 5.41×R, under the cooling rate of 200 °C/s. Fig. 9(d) indicates the intensity of \{111\} \(<112>\> and \{111\} \(<110>\> texture components decreased by 9.8% and 6.5% under the cooling rate of 500 °C/s, compared with those formed under the cooling rate of 200 °C/s. The intensity of \{011\} \(<100>\> orientation decreased a lot, from 1.77×R to 1.05×R. Finally, when the cooling rate was 1000 °C/s, the intensity of \{111\} \(<110>\> and \{011\} \(<100>\) components increased, while the intensity of \{111\} \(<112>\> decreased.
Fig. 9 ODFs of the IF steel annealed under different cooling rates

(a) 5 °C/s; (b) 50 °C/s; (c) 200 °C/s; (d) 500 °C/s; (e) 1000 °C/s
4. Discussion

4.1 Effects of cooling rates on mechanical properties

The mechanical properties of samples annealed under different cooling rates are shown in Fig. 11. The yield strength and $r_{\text{ave}}$ values increase with increasing cooling rates, except that the sample annealed under the cooling rate of 500 $^\circ$C/s possesses lower $r$ value (2.21). The elongation rates, $n$ values and UTS change little under different cooling rates.
According to the measured mechanical properties, the yield strength increases as the increase of cooling rates. The grain boundary strengthening mechanism is usually described by the Hall-Petch equation as follows:

$$\sigma_y = \sigma_0 + k_y d^{-1/2}$$  \hspace{1cm} (4-1)

where \(\sigma_y\) is the yield strength, \(d\) is the average grain diameter, \(\sigma_0\) is the friction stress and \(k_y\) is the Hall-Petch slope[23]. According to the grain sizes obtained under different cooling rates, the grain diameters decrease as the increase of cooling rates. The grain size of the sample annealed under the cooling rate of 5 \(^\circ\)C/s is 15.2\(\mu\)m, while under the cooling rate of 1000 \(^\circ\)C/s, the value decreases to 11.7\(\mu\)m. Based on the Hall-Petch equation, the decrease of grain diameters will result in the increase of yield.
strength. This is consistent with the increase of yield strength values as the increasing of cooling rates in this paper.

The little change of UTS, elongation and n values can be explained as follows. Solution strengthening is the major method to increase UTS in this IF steel. All the samples contain the same amount of P as solution strengthening atoms and thus the UTS changes little in different steels. Ferrite structure and large grains are responsible for retaining the ductility in low carbon steels[19, 29]. Since all samples are composed of 100% ferrites and the difference of grain sizes formed under the cooling rates from 5 °C/s to 1000 °C/s is small, the elongation changes little. Strain hardening exponent (n) is an important parameter to assess the deformation characteristic of steel and it depends mainly on the structure types in low carbon steels[19]. The ferrites lead to more hardening by large amount of deformation compared with other structure types. All samples formed under different cooling rates are all made up of ferrite structure explains the little change of n values in this paper.

4.2 Precipitation behavior

According to the second phase solid solubility formula in steels, the TiN and TiC solid solubility formulas based on phase analysis are[30]

\[ \lg \{[\text{Ti}][\text{N}]\}_\gamma = 3.94 - 15190 / T \]  \hspace{1cm} (4-2)

\[ \lg \{[\text{Ti}][\text{C}]\}_\gamma = 5.33 - 10475 / T \]  \hspace{1cm} (4-3)

Their precipitation temperatures are 1355 °C and 872 °C respectively based on these two formulas. The Ti compounds precipitation temperature has been studied and previous researchers has made schematic illustration regarding the sequence of precipitates formation and their stability[31]. According to his research, TiN takes
place at 1300 °C, while TiC and FeTiP at about 700 °C. It is also found that TiN is more stable than TiC in their studies. Park has pointed out that some oxides can act as heterogeneous nuclei for the formation of TiN in ferritic chromium during steel making route[31]. Some TiN precipitation took place in liquid based on previous studies and the process is controlled by solubility limit of Ti-N equilibrium. CaO and SiO\(_2\) are slag particles and they formed at liquid stage. These oxides acted as nucleation for TiN precipitates at high temperatures and these TiN present in both all the cooling rates.

The formation of large size TiN (shown in Fig. 5), which has oxide nuclei, are due to they have more time to grow under the common cooling rates. In order to explain the different sizes of pure TiN formed under different cooling rates, diffusion controlled growth mechanism needs to be considered. Free standing TiN forms during re-heating and hot rolling and it is the interstitial nitrogen which is trapped first by Ti and remained so. As for free standing TiC, they usually take place during coiling. Comparing the sizes of pure TiN in non-annealed sample and the sample annealed under the cooling rate of 1000 °C/s, we can learn that TiN grows during the holding section. As the cooling rates decrease from 1000 °C/s to 5 °C/s, the pure TiN particles have obvious tendency to grow bigger. The cooling rates may change the dimensions growth number and ultimately affects the time exponents and transformation process[32,33].

Apart from these, FeTiP precipitates are observed under the cooling rate of 5 °C/s. The precipitation behavior of FeTiP has been studied before. In batch annealing, the
size of FeTiP precipitate is big and the content of phosphorus atoms is high[2]. On the contrary, in the continuous annealing process, FeTiP is observed on the pre-existing TiC and the content of phosphorus is low[3]. It has been reported that FeTiP takes place over TiC particles, suggesting that FeTiP forms only after the formation of TiC[2]. This is consistent with the FeTiP precipitation behavior in this paper. It is reported that phosphorus accumulates at the bottom of the edge dislocation since the radius of phosphorus (0.109 nm) is smaller than that of α-Fe (0.124 nm)[13]. This is illustrated in Fig.12. Phosphorus atoms are driven by moving dislocation lines and accumulate at grain boundaries during continuous annealing process[13]. The FeTiP precipitates are found near TiC particles (Fig.6) in this study. Based on the above conclusions, we can assume that phosphorus, which concentrates at grain boundaries, interacts with Ti and Fe atoms. Thus FeTiP forms on pre-existing TiC particles. This mechanism of FeTiP formation is consistent with the previous result that FeTiP may not be a strict stoichiometric compound [11]. It has been studied that the formation of FeTiP precipitates strongly prevents the growth of {111} recrystallization texture which is beneficial for deep-drawing property[2, 3].

Fig. 12 Schematic of phosphorus and edge dislocation
4.3 Effect of texture on r value

All steels possess high $r_{ave}$ values except those annealed under the cooling rates of 5 °C/s and 500 °C/s (2.13 and 2.21 respectively). It is well known that the forming property can be characterized by the $r_{ave}$ and $\Delta r$ values which are strongly affected by the texture components. In order to get good forming property, the material should have high $r_{ave}$ value and low $\Delta r$ value[34, 35]. D. Daniel[35] has studied the relationship between r values and texture components in low carbon steels and the statistics are listed in Tab.6. It can be learnt that the {111} <110>, {111} <112> and {011} <001> components are the most favorable to increase the r value, so the intensity of these three components formed under different cooling rates is plotted in Fig.13. From Tab.6, we can learn that the {011} <001> component contributes most to the increase of r value, but it also increases $\Delta r$ value dramatically. The {111} <110> and {111} <112> components can increase r value significantly without increasing $\Delta r$ value. Different texture components correspond to different $r_{ave}$ and $\Delta r$ values. In addition to texture components, Chung[24] suggests that grain morphology also has an effect on the mechanical property anisotropy and equiaxed grains normally correspond to the low $\Delta r$ value. Since all samples are made up of equiaxed ferrite grains and the difference of average grain aspect ratio is small, the effect of grain morphology on $\Delta r$ value could be ignored.
Tab. 6 The correlation between texture components and \( r \) values in BCC materials

<table>
<thead>
<tr>
<th>Texture Components</th>
<th>( r )</th>
<th>( \Delta r )</th>
</tr>
</thead>
<tbody>
<tr>
<td>{001}&lt;110&gt;</td>
<td>0.4</td>
<td>-0.8</td>
</tr>
<tr>
<td>{112}&lt;110&gt;</td>
<td>2.1</td>
<td>-2.7</td>
</tr>
<tr>
<td>{111}&lt;110&gt;</td>
<td>2.6</td>
<td>0</td>
</tr>
<tr>
<td>{111}&lt;112&gt;</td>
<td>2.6</td>
<td>0</td>
</tr>
<tr>
<td>{554}&lt;225&gt;</td>
<td>2.6</td>
<td>1.1</td>
</tr>
<tr>
<td>{011}&lt;001&gt;</td>
<td>5.1</td>
<td>8.9</td>
</tr>
</tbody>
</table>

The FeTiP precipitate was observed under the cooling rate of 5 °C/s and its formation result in the weak intensity of \{111\} <112> and \{111\} <110> components (4.09×R and 4.15×R respectively). According to the analysis, the \{111\} <110> and \{111\} <112> components are favorable to increase \( r \) values. Then the weak texture components of \{111\} <112> and \{111\} <110> are responsible for the low \( r_{ave} \) value (2.13) in the sample annealed under the cooling rate of 5 °C/s.

The sample annealed under the cooling rate of 500 °C/s also has a lower \( r_{ave} \) value (2.21) and lower \( \Delta r \) value (0.75) compared with those annealed under the cooling rates of 200 °C/s and 1000 °C/s. As shown in the Fig.13, we can learn that the sample annealed under 500 °C/s has the weakest \{011\} <001> fiber. According to the relationship between texture components and \( r \) value, the strong \{011\} <001> component significantly contributes to the increase of \( r \) and \( \Delta r \) value. Thus, the lower \( r \) and \( \Delta r \) value in the sample annealed under the cooling rate of 500 °C/s is attributed to the weak intensity of \{011\} <001>.

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5. Conclusions

(1) The yield strength, \( r \) and \( \Delta r \) values are strongly influenced by cooling rates. The yield strength increases as increasing of cooling rates from 5 °C/s to 1000 °C/s. The \( r \) values also show a rising trend as the increase of cooling rates, except that the sample annealed under the cooling rate of 500 °C/s possesses lower \( r \) value. In addition, in the case of 500 °C/s, the sample also has the lowest \( \Delta r \) value. The ultimate tensile strength, \( \sigma \) values and elongation rates change little under all different cooling rates.

(2) The size of pure TiN precipitates decreases as increasing of cooling rates. The oxides, like CaO and SiO\(_2\), act as heterogeneous nuclei for TiN and these TiN have larger size under the cooling rates of 5 °C/s and 50 °C/s. FeTiP can be observed on the pre-existing TiC particles under the cooling rate of 5 °C/s and the presence of FeTiP precipitate prevents the growth of recrystallization \{111\} texture.
(3) The intensity of \{111\} <110> orientation increases as the cooling rate increases from 5 °C/s to 1000 °C/s while the intensity of \{011\} <100> decreases as the cooling rate increases from 5 °C/s to 500 °C/s. There is a texture transition in the γ-fiber: from \{111\} <112> to \{111\} <110> under the cooling rate of 1000 °C/s. The \( r \) and \( \Delta r \) values have strong connection with texture components. The low \( r_{\text{ave}} \) under the cooling rate of 5 °C/s is attributed to the weak \{111\} <112> and \{111\} <110> components. The weak \{011\} <100> orientation is responsible for the low \( r_{\text{ave}} \) and \( \Delta r \) values under the cooling rate of 500 °C/s.

References:


Highlights:

(1) The best formability steel can be obtained under the cooling rate of 200 °C/s.

(2) FeTiP precipitates on parts of pre-existing TiC under the cooling rate of 5 °C/s.

(3) The size of pure TiN decreases as increasing of cooling rates.

(4) The intensity of \{011\} <110> orientation decreases with increasing cooling rates.

(5) The weak \{011\} <100> orientation is responsible for low r and ∆r values.